WAR FOOD ADMINISTRATION

OFFICE OF MARKETING SERVICES

METHODS EMPLOYED IN THE LABORATORY ANALYSIS OF SWEETENED CONDENSED MILK BY THE DAIRY AND POULTRY BRANCH

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THE CHEMICAL ANALYSES OF SWEETENED CONDENSED MILK

Sweetened condensed milk is analyzed with a Mojonnier Tester using methods which are essentially the same as those recommended by the Mojonnier Bros. in their Manual.

Determination of Net Weights

A composite of sweetened condensed milk representing 3000 cases is made up as follows:

The odd numbered cans of milk are weighed to an accuracy of 0.01 ounce; these cans are opened, emptied into an adequate container, and poured back and forth at least four times, using another similar sized container. Approximately two ounces of the well mixed sample is taken for testing and the remainder of the milk is discarded. The empty cans are washed in plain tap water without soap, dried and reweighed to obtain the net weight. If the average weight of the odd-numbered cans is less than 15.0 ounces (or whatever the contract weight should be), the even-numbered cans are also weighed. It is observed that only half of the cans are originally used for compositing. In the event of the milk being below standard in fat, solids, sucrose or net weight, the remaining cans may be employed to determine which of the individual lots did not meet specifications.

Solids Determination

With the aid of a pipette distribute evenly approximately 1.0 gm. of well mixed sweetened condensed milk in a previously tared covered aluminum "solids dish." Add 3 or 4 ml. hot distilled water; warm mixture to approximately 80° C and shake with rotary motion until sample is completely dissolved. Place dish on 180° C "hot plate" and carefully evaporate the mixture to a uniform light tan shade. Transfer dish to the "solids oven" at 100° C, heating for 90 minutes with not less than 20" vacuum. Place in desiccator and cool to room temperature (7 to 10 minutes) reweigh, and calculate as percentage of total solids in the sample.

Butterfat Determination

Transfer into a Mojonnier flask approximately 5.0 gms. of the well mixed composite of sweetened condensed milk (using weighing pipettes and holder) add 7 ml. of hot distilled water and mix. Add 1.5 ml. of 28% ammonium hydroxide. The contents are again well mixed and allowed to cool. Add 10 ml. of ethyl alcohol and mix thoroughly. Three drops 0.5% phenolphthalein solution are added to more clearly define the ether layer from the residue. Add 25 ml. ethyl ether, stopper and shake vigorously. Next add 25 ml. of petroleum ether, stopper, and again mix by vigorously shaking. With a hand centrifuge, rotate the flask 60 times in a period of 1 minute. Decant the

clear ether layer into a weighed aluminum fat dish (high side walls) and evaporate the ether slowly on a hot plate (135° C). The temperature should be sufficient to allow complete evaporation, but not so high that spattering or vigorous boiling will result.

Make a second extraction using 5 ml. of ethyl alcohol (instead of 10 ml. as for the original extraction) and the same quantities of each ether; mix well after the addition of each reagent. Centrifuge 60 times, decant the clear ether layer into the corresponding aluminum dish and evaporate slowly. If necessary, carefully pour a few ml. of distilled water down the side of the flask just prior to pouring off the second extraction to raise the level of the aqueous layer, so ethers may be completely poured off. It is important that none of the aqueous layer be allowed to run into the aluminum dish. After the ether is entirely evaporated from the aluminum dish on the "hot plate", place it in the Mojonnier oven for 5 minutes with the temperature at exactly 135° C and a vacuum of not less than 20". Transfer and cool to constant weight in the cooling desiccator. In weighing the dishes, both when empty and when containing the extracted fat, they should be at room temperature. This usually requires cooling in the Mojonnier desiccator for 7 to 10 minutes. Report as percent fat.

When the original composite sample is found to be below Government standards for fat, a recheck is made using three extractions.

Determination of Sucrose (Modified Mojonnier Method using Polariscope Reading in the Ventzke Scale

Weigh 26.048 gms. of well mixed sweetened condensed milk sample into a beaker. With the aid of approximately 70 ml. of hot water transfer the milk into a 100 ml. volumetric flask washing down the sides of the beaker and flask. The flask is then placed in a hot water bath (95-100° C) for a period of 15 minutes. Remove flask and allow to stand overnight at room temperature to destroy mutarotation.

Add 4 ml. of acid mercuric nitrate,(1) dilute to volume and then add an excess of 4 ml. water. Shake vigorously for 2 minutes, filter and fill polariscope tube with some of the filtrate. The reading on the polariscope is taken at about 20° C within 10 minutes after adding the mercuric nitrate solution. This is the "direct reading" in Clerget's formula.

50 ml. of the above filtrate are transferred into a 100 ml. flask and 5 ml. of 1:1 HCl added for the inversion of the sucrose. Place in a 70° C water bath for 10 minutes, cool, filter if necessary, and then transfer a portion to the polariscope tube for the "invert reading." The direct and invert readings should be made at the same temperature; however, if both readings are not exactly at the same temperature use the invert reading temperature in the Clerget's formula. Correct for volume $\frac{55}{50}$ x invert reading.

(1) The Acid Mercuric Nitrate Reagent is prepared as follows:
To 110 gms. yellow HgO carefully add 150 ml. of distilled water and sufficient concentrated HNO_3 (80 ml.) to form a clear solution, being careful to use least possible excess of acid. Dilute to 300 ml. and add 30 ml. 5% NaOH slowly. Heat for 2 minutes, cool, dilute to 500 ml. and filter.

The percent of sucrose is obtained by the use of Clerget's formula:

$$S = \frac{100}{142.68 - \frac{T}{2}} \quad (D - I)$$

Where S = percent sucrose, D = "direct reading", I = "invert reading" and T = temperature in degrees C.

If percent lactose is desired use formula L = (D-S) x 1.266

Alternative Gravimetric Method for Sucrose (Polariscope not required)

The method outlined is a modification of a gravimetric procedure described by White ("The Technical Control of Dairy Products" by Mojonnier and Troy, 1925).

Ten grams of the well mixed sweetened condensed milk sample are weighed into a 50 ml. beaker. With the aid of about 125 ml. of hot distilled water transfer the milk into a 250 ml. volumetric flask, washing down the sides of the beaker and the flask. Shake contents of flask and cool to room temperature. Add 12 ml. of Fehlings A (copper sulfate) and 6 ml. N/2 NaOH; shake and dilute to volume. Add 1.5 ml. of distilled water from a dilution pipette to allow for the precipitated protein. Filter, discarding the first few ml. of filtrate. If all of the protein is precipitated the filtrate should have a blue cast; the amount of Fehlings A must be increased if filtrate is colorless.

Lactose Determination: Transfer 25 ml. of each of Fehlings A and B in a 400 ml. beaker and add 50 ml. of the filtrate prepared as above. Place watch glass over beaker and heat on asbestos gauze over Bunsen burner, regulate the flame so that boiling begins in 4 minutes, and continue boiling for exactly 2 minutes. It is important that these directions be strictly observed. To regulate burner for this purpose it is advisable to make preliminary tests, using 50 ml. of the reagent and 50 ml. of water before proceeding with the actual determination. Keep beaker covered with watch glass during entire heating.

Filter hot solution immediately thru a conditioned "Selas" 0.01 porosity crucible using suction. Wash precipitate of Cu_2O thoroughly with not more than 75 ml. hot water. If lactose percentage is desired, wash the Cu_2O thoroughly with alcohol and ether. Weigh as Cu_2O (or determine the copper directly). With the use of Munson and Walker's table read the number of milligrams of lactose equivalent to the weight of Cu_2O. If copper is determined directly follow procedure outlined on page 4 under "Copper determination by titration." Calculate percentage of lactose in sweetened condensed milk.

Invert Sugar Determination: The filtrate is transferred to a 250 ml. volumetric flask. Add 9 ml. 1:1 HCl to neutralize filtrate and an additional 25 ml. of acid for the inversion. Heat rapidly to 70° C in a water bath with constant stirring to prevent overheating any part of the liquid. Hold in a water bath of 70° C for 45 minutes. (Best results are obtained by not allowing the temperature of the water bath to exceed 71° C). Cool. Neutralize with 1:1 NaOH using a few drops of phenolphthalein solution as an indicator. Generally the characteristic blue color is noted near the neutral point. If the solution is basic it should immediately be brought back to the neutral

point or slight acid with 1:1 HCl before the sugar is allowed to precipitate the copper from solution. Cool to room temperature and dilute to 250 ml. mark and thoroughly mix. Pipette 50 ml. of the invert solution and transfer to a 400 ml. beaker in which had previously been placed 25 ml. each of Fehlings A & B. Place watch glass over beaker and heat over the same regulated flame that will bring the solution to a boil in 4 minutes. Boil for 2 minutes and filter immediately through a Selas crucible. Wash with hot water at least three times.

Copper determination by titration: Because of the impurities it is advisable to determine the copper directly by titration rather than to weigh as Cu_2O. The Selas crucible containing the Cu_2O is placed in a 400 ml. beaker and enough concentrated HNO_3 carefully added to fill the crucible to about three-fourths full. Stir, then add sufficient distilled water to fill the crucible and stir again. Empty the crucible carefully and fill half full with conc. HNO_3 and the remainder with 1:1 HCl. Stir, empty contents of the crucible into beaker and wash carefully with distilled water.

The bottom of the crucible should be white with no traces of blue or red. If it is not white continue washing with acid and water until all of the copper is dissolved. Then add 10 ml. 1:1 H_2SO_4 to the solution and wash down the sides of the beaker. Boil under hood to expel fumes of NO_2 and until solids start to precipitate. Cool, wash down the sides of the beaker and watch glass with hot distilled water. Neutralize with NH_4OH (with agitation) until a faint turbidity of $Cu(OH)_2$ appears. If the end point is passed and the blue of the amine is observed, add a few drops of 1:8 H_2SO_4. Then add an excess of 5cc 1:8 H_2SO_4.

Add 2.0 gms. of KI and stir well for 30 seconds. Titrate at once with the sodium thiosulfate solution until the brown color becomes faint; then add 5 ml. of starch indicator and titrate to a cream color. The solution of sodium thiosulfate is so prepared that 1 ml. is equivalent to 0.01 gms. of copper.

Standard thiosulfate solution: 19.50 gms. of sodium thiosulfate (Mallinckrodt or Merck analytical reagent) are dissolved and diluted to 500 ml. This solution should be prepared fresh each week. Next weigh accurately 0.3000 gms. of pure copper and transfer to a beaker. Dissolve the copper in about 5 ml. concentrated HNO_3. Add 5 ml. of 1:1 H_2SO_4 and boil under the hood until fumes of NO_2 are completely driven off. Cool, wash down the sides of the beaker and watch glass with hot distilled water. Neutralize with NH_4OH (with agitation) until a faint turbidity of $Cu(OH)_2$ appears. Follow the same procedure as outlined above.

Calculations:

(Mg of copper) - 1.1 (lactose correction factor) = net mg of Copper (due to invert sugar). From Munson and Walker tables find the mg of invert sugar.

$$\text{Percent sucrose} = \frac{\text{mg. invert sugar } (0.95)}{\text{wt. of sample } (400 \text{ mg})} \times 100$$

Reagents

Starch Solution - Shake up one gram of soluble starch (U.S.P.grade) with about 10 ml. cold water and pour this suspension into 200 ml. boiling water. Continue heating for a few minutes. Cool, dilute to 500 ml. (Preserve with metallic Mercury) Starch solutions cannot be kept for more than a day or two unless a preservative is added.

Fehlings A - Dissolve 69.278 gms. of copper sulfate in water, dilute to 1 liter, and filter

Fehlings B - Dissolve 346 gms. of Rochelle salts (Potassium sodium tartrate) and 100 gms. of NaOH in H_2O, dilute to 1 liter. Allow to stand overnight and filter.

Potassium Iodide - U.S.P. Fine crystals.